

## EXPERIMENTAL INVESTIGATION OF THERMAL STABILITY OF CARBON NANOTUBES REINFORCED ALUMINIUM MATRIX USING TGA-DSC ANALYSIS

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### ABSTRACT

*Metal matrix composites are grabbing more attention by researchers due to their outstanding properties which meet the requirements of a specific application. This paper deals with the experimental investigations done to examine the thermal stability of Matrix under various thermal applications and also to study the effect of MWCNT content and particle size on thermal properties of the Al-CNT metal matrix. Different characterization techniques such as Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry analysis (DSC) were performed for the Aluminium-CNT metal matrix. Thermal gravimetric analysis revealed that maximum weight loss is found to be 0.44% for 1.5 wt % of CNT (type2) in Aluminium which is less compared to the weight loss of pure Aluminium. Hence a minimum of 0.24% weight loss was found for 1.0 weight % of CNT (type 2) in Aluminium. Differential Scanning Calorimetry analysis revealed that rate of heat flow increase gradually with the increase in outer diameter of selected carbon nanotubes and maximum heat flow of 106.6 mW for 1.5 wt% of CNT (type3) in Aluminium which was 40% greater than that of pure Aluminium. Thus TGA-DSC tests help in investigating the thermal stability of selected Al-CNT metal matrix.*

**KEYWORDS:** Carbon nanotubes (CNT), Powder Metallurgy, Metal matrix composites, ThermoGravimetric Analysis (TGA) & Differential Scanning Calorimetry (DSC)

**Received:** Feb 20, 2018; **Accepted:** Mar 13, 2018; **Published:** Apr 27, 2018; **Paper Id.:** IJMPERDJUN201818

### INTRODUCTION

Carbon nanotubes (CNTs) have transpired as materials with freakish properties exceeding those of any typical material from the time of their discovery [1]. However, the main research shove over the past decade has focused on applications of CNTs for reinforcing polymer and ceramic matrices a few groups have investigated metallic matrices with the core interest being pure aluminum [2-3]. Defect-free CNTs, both single-walled (SWNTs) and multi-wall (MWNTs) have elastic moduli of 1 TPa and tensile strengths in the region of 150 GPa can be a good material for structural materials.[4] Carbon nanotubes possess high thermal conductivity and good thermal stability

which increases gradually with increase in diameter. [5] In fact, the interest in CNT reinforced metal matrix composites has been growing considerably. Various researchers have focussed on the production of CNT-Metal matrices with enhanced thermal properties. Such composites would make attractive novel materials with potential applications in the aerospace, automotive and sports industries. Aluminum is the most used metal after steel, as the density of aluminum is approx. 2.6 to 2.8 g per cm<sup>3</sup>. Use of aluminum for domestic purposes is economical, safe and ecologically sensible. The oxidation and ignition characteristics of aluminum particles have been widely investigated in the past decade [6]. However, in order to have more controllability over the material's properties, aluminum is usually alloyed by mixing with several other elements at the bulk scale. Due to the synergistic effects of the constituent alloying materials, the physiochemical properties of the alloys can be improved by chemical reordering and spatial redistribution of the atoms [7]. Aluminum is one of the best choices for replacing many metals in the field of automobiles due to its corrosion resistance capability and good formability. Hence aluminum alloys are being fabricated with the addition of various elements like carbon nanotubes and other nano-sized particles so as to enhance the properties of metal matrix to meet the specific applications [8]. One of the major applications is automobile sector wherein the engine piston should be designed with materials of high strength and low thermal expansion to withstand the high combustion pressure and temperature [9-11]. Thermogravimetric analysis, (TGA) is a technique used to monitor the mass of any substance subjected to increment/decrement in temperature. Differential Scanning Calorimetry (DSC) analysis measures the amount of energy absorbed or released by a sample when it is heated or cooled, providing quantitative and qualitative data on endothermic (heat absorption) and exothermic (heat evolution) processes. Thus TGA-DSC tests help to understand and examine the thermal stability of metal matrix composites subjected to thermal loading conditions under various applications like automobile engine blocks, heads and heat exchangers etc.,

## EXPERIMENTATION

### Selection of Materials

As received Aluminium powder is 99% pure with a mesh size of minimum 90 % (passing through 200 meshes) and the Multi-Walled Carbon nanotubes are selected as three types with the specifications mentioned in Table 1.

**Table 1: Specifications of Selected MWCNT**

Specifications	Type-I	Type-II	Type-III
Length (μm)	10 – 30	10 - 30	10 - 30
Outer Diameter (nm)	< 8	10 - 20	30 - 50
Purity (%)	Min 95	Min 95	Min 95

### Fabrication of Al-CNT Metal Matrix

The Fabrication of Al-CNT metal matrix composites is done through Powder Metallurgy process which involves different steps in it. The different compositions of Al-CNT powder are subjected to ball milling process in order to have uniform mixing of CNT in Aluminium powder and to maintain rigid bonding between the two powders. After achieving the Nano sizes the next process is to compact by pressing under a high pressure and load. Compaction is done generally to make the bonding of powders and get adhered to each other. The holding time was 10seconds. After that, the plunger moves upward and the required solid shapes were achieved. The obtained specimens are heated in a controlled-atmosphere furnace to a temperature below its melting point. These specimens are kept in the furnace for nearly 2hours and dwell time of 2hours cooling to room temperature. The temperature and voltage are set in the digital indicators. The specimens are removed from the furnace.

### Thermo Gravimetric Analysis (TGA)

Different types of TGA analysis are available viz. Isothermal or static TGA, Quasi-static TGA, and Dynamic TGA. In the present work, the dynamic TGA was performed wherein the sample is subjected to the condition of a continuous increase in temperature at a constant heating rate, i.e., usually linear with time. A sample of 30 mg was collected from all the specimens and placed in a pan made of Alumina ( $\text{Al}_2\text{O}_3$ ). The specimens were characterized under inert atmosphere during heating to a pre-determined temperature of  $600^\circ\text{C}$  at a heating rate of  $20^\circ\text{C min}^{-1}$  and in the temperature range of  $600^\circ\text{C}$  to  $700^\circ\text{C}$  the heating rate was set at  $5^\circ\text{C min}^{-1}$  to keep a continuous record as it reaches the melting point of aluminum. The specimen was allowed to stabilize for 15 min at melting temperature and later it was allowed to reach room temperature.



**Figure 1: Experimental Setup used to Perform TGA-DSC Analysis**

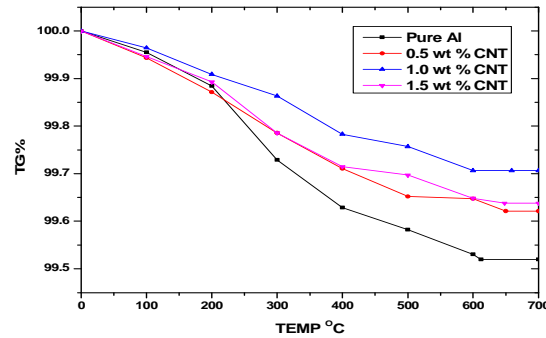
### Differential Scanning Calorimetry (DSC) Analysis

DSC analysis measures the amount of energy absorbed or released by a sample when it is heated or cooled, providing quantitative and qualitative data on endothermic (heat absorption) and exothermic (heat evolution) processes. A sample of 30 mg was collected from all samples made of different compositions of CNT in Aluminium matrix. The sample is placed on a pan made of Alumina ( $\text{Al}_2\text{O}_3$ ). The specimens were characterized under inert atmosphere during heating to a temperature of  $700^\circ\text{C}$ . The rate of heat flow was observed closely when the temperature reaches the melting point of aluminum.

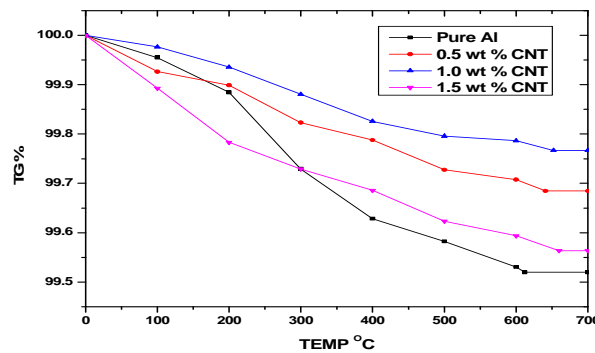
## RESULTS AND DISCUSSIONS

Thermo Gravimetric analysis was employed to examine the percentage weight loss of as-prepared Al-CNT metal matrix for different compositions and different types of CNTs. It was observed that TGA curves decrease steadily from the start without any exothermic or endothermic reaction. The thermogravimetric analysis of Al-CNT (type1) metal matrix from figure 2 shows a maximum weight loss of 0.38% for 0.5 weight % of CNT in Aluminum and minimum weight loss of 0.29% for 1.0 weight % of CNT in Aluminum where in pure Aluminum showed the weight loss of 0.49 %. Similarly the thermogravimetric analysis of Al-CNT (type2) metal matrix from figure 3 shows a maximum weight loss of 0.44% for 1.5 weight % of CNT in Aluminum and minimum weight loss of 0.24% for 1.0 weight % of CNT in Aluminum and Al-CNT (type3) metal matrix from figure 4 shows a maximum weight loss of 0.38% for 1.5 weight % of CNT in Aluminum and minimum weight loss of 0.27% for 0.5 weight % of CNT in Aluminum respectively. Differential Scanning Calorimetry (DSC) Analysis was performed to measure the rate of heat transfer capacity of Al-CNT metal matrix. The DSC curves for all the specimens were found to be constant until the melting point temperature and showed a downward peak indicating

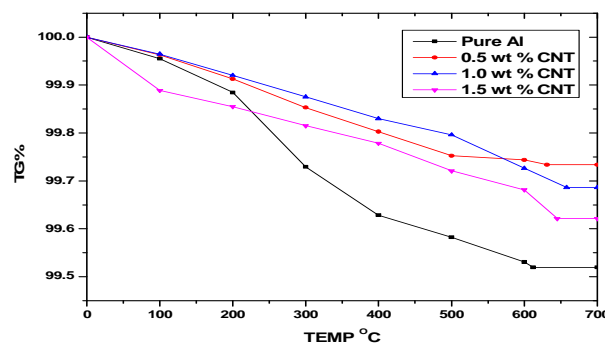
the maximum heat flow at the transition temperature. Figure (5 to 7) shows the DSC curves for Al-CNT (type1) metal matrix where in a maximum heat flow of 76.5 mW was recorded for 1.0wt% of CNT in Aluminum matrix. Similarly Figure (8 to 10) shows the DSC curves for Al-CNT (type2) metal matrix with maximum heat flow of 80.3 mW for 1.0wt% of CNT in Aluminum matrix and Figure (11 to 13) shows the DSC curves for Al-CNT(type3) metal matrix with maximum heat flow of 106.6 mW for 1.5wt% of CNT in Aluminum matrix respectively.



**Figure 2: Thermogravimetric Analysis of Al-CNT (Type-1) Metal Matrix for Different Compositions of MWCNT: Indicates the Percentage Weight Loss of Matrix due to Increase in Temperature**



**Figure 3: Thermogravimetric Analysis of Al-CNT (Type-2) Metal Matrix for Different Compositions of MWCNT: Indicates the Percentage Weight Loss of Matrix due to Increase in Temperature**



**Figure 4: Thermogravimetric Analysis of Al-CNT (type-3) Metal Matrix for Different Compositions of MWCNT: Indicates the Percentage Weight Loss of Matrix due to Increase in Temperature**

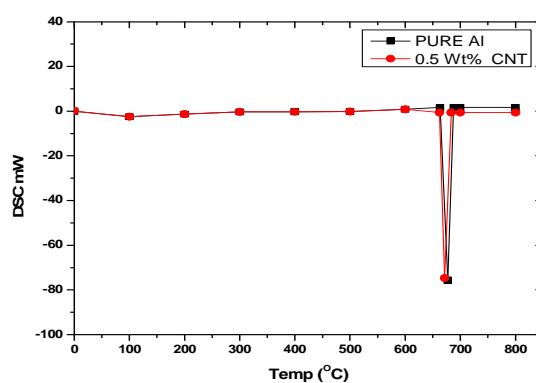


Figure 5: DSC Analysis of Al+0.5wt% CNT (Type1): Comparison of Rate of Heat Flow/Transfer with that of Pure Aluminium

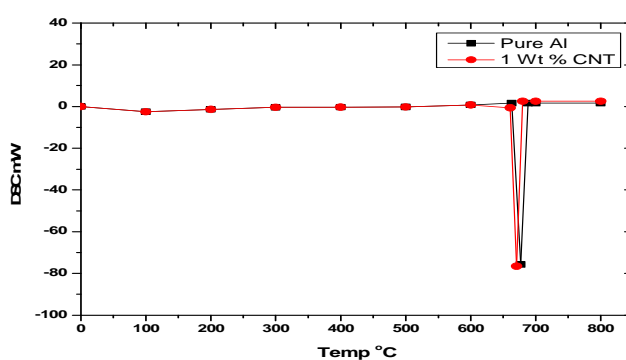


Figure 6: DSC Analysis of Al+1wt% CNT (Type1): Comparison of Rate of Heat Flow/Transfer with that of Pure Aluminium

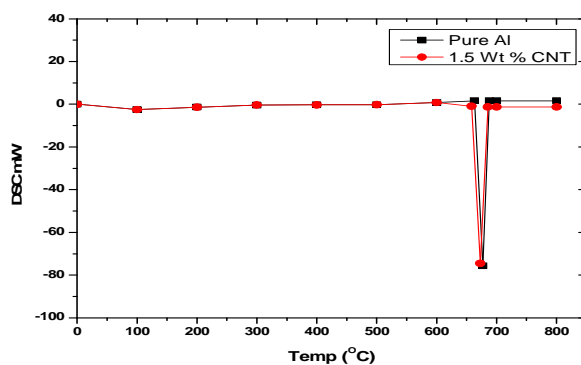
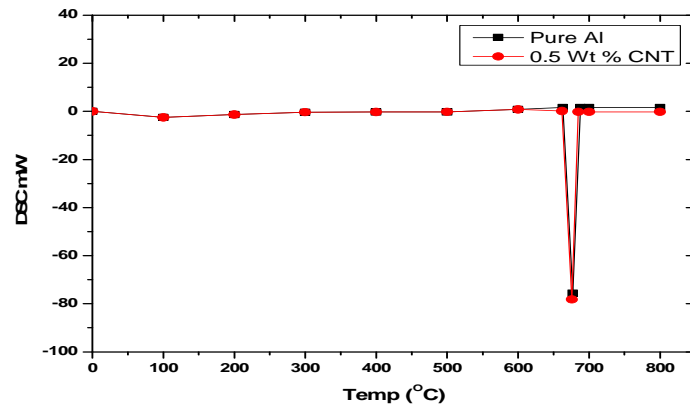
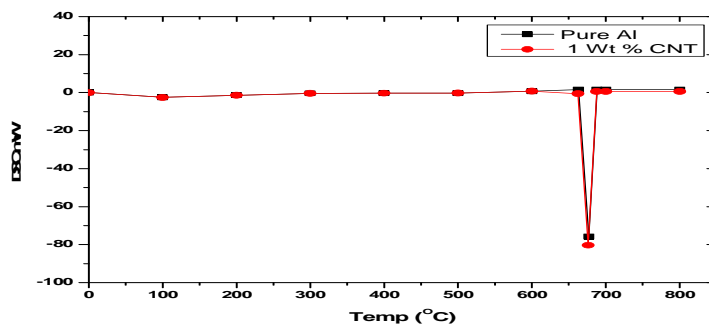


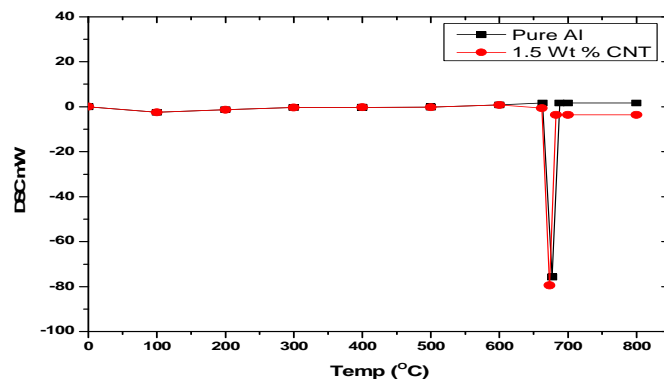
Figure 7: DSC Analysis of Al+1.5wt% CNT (Type1): Comparison of Rate of Heat Flow/Transfer with that of Pure Aluminium



**Figure 8: DSC Analysis of Al+0.5wt% CNT (Type2): Comparison of Rate of Heat Flow/Transfer with that of Pure Aluminium**



**Figure 9: DSC Analysis of Al+1wt% CNT (Type2): Comparison of Rate of Heat Flow/Transfer with that of Pure Aluminium**



**Figure 10: DSC Analysis of Al+1.5wt% CNT (Type2): Comparison of Rate of Heat Flow/Transfer with that of Pure Aluminium**

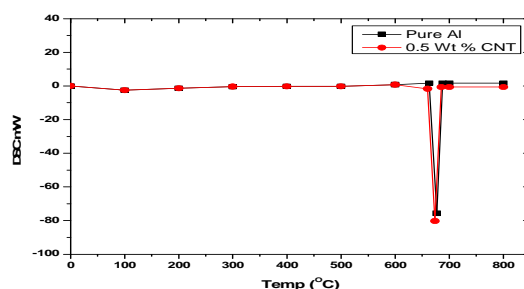


Figure 11: DSC Analysis of Al+0.5wt% CNT (Type3): Comparison of Rate of Heat Flow/Transfer with that of Pure Aluminium

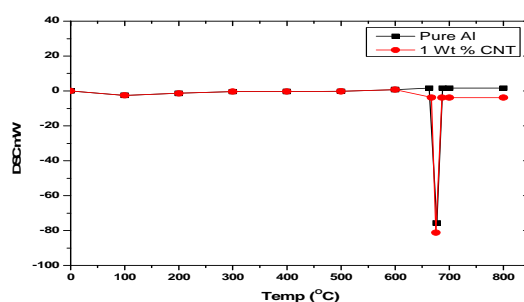


Figure 12: DSC Analysis of Al+1wt% CNT (Type3): Comparison of Rate of Heat Flow/Transfer with that of Pure Aluminium

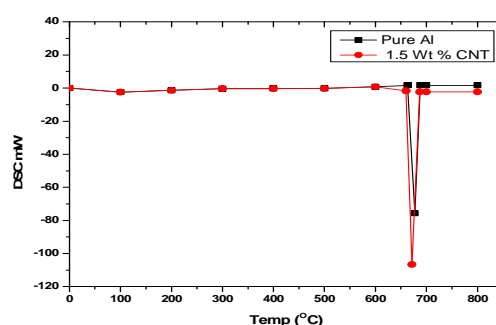


Figure 13: DSC Analysis of Al+1.5wt% CNT (Type3): Comparison of Rate of Heat Flow/Transfer with that of Pure Aluminium

## CONCLUSIONS

The TGA-DSC results revealed that the thermal stability of Al-CNT metal matrix can be increased with the addition of carbon nanotubes. The percentage weight loss of Al-CNT metal matrix subjected to heating was less compared to pure aluminum. The rate of heat flow of Al-CNT metal matrix increased gradually with increase in CNT content and also with the increase in outer diameter of CNT (i.e., type3). Hence due to good thermal stability, this metal matrix composites can be used as an alternative to metals in most of the thermal applications like automobile engine blocks, heads, and heat exchangers.

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